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पॉलीकार्बोनेट संचकन और बहिर्वेधन सामग्री — विशिष्टि

Indian Standard

POLYCARBONATE MOULDING AND EXTRUSION MATERIALS — SPECIFICATION

ICS 83.080.20

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

April 1998 **Price Group 10**

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Plastics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Since commercialization of polycarbonate about a quarter century back, it has established itself as one of the most versatile engineering thermoplastic material. This versatility of polycarbonate emanates from excellent combination of mechanical strength, temperature resistance, electrical insulation, optical clarity, wide usability temperature range and for its virtual unbreakability. Industries those are being catered by polycarbonate for variety of application include automobile, business machines, electrical and electronics, lighting, telecommunication, appliances, safety and security, food and medical, leisure and building and construction.

In India, till couple of years back, the use of polycarbonate was very limited. But with the national drive to upgrade our industries, the consumption of polycarbonate is now growing steadily and has already reached a respectable level. There is only one indigenous manufacturing facility for polycarbonate compounds as on date and most of the country's requirement is fulfilled by this facility. The wide spectrum of industries those are now using polycarbonate are not only anxious about steady supply of material but also they are feeling the need of Standard to fall back upon while procuring it to ensure maintenance of quality of their products. These considerations led the concerned Technical Committee to formulate this Indian Standard on polycarbonate moulding and extrusion materials.

Since polycarbonate is relatively new to the processing community in India and performance of parts moulded from polycarbonate is affected by adoption of improper processing techniques, it was thought prudent to include a code of good processing practices of polycarbonate in this standard (*see* Annex A).

Considerable assistance has been derived from the following publications while preparing this standard:

ISO 7391-1 : 1987 'Polycarbonate moulding and extrusion materials — Part 1 Designation'. Issued by the International Organization for Standardization (ISO).

ISO 7391-2 : 1987 'Polycarbonate moulding and extrusion materials — Part 2 Preparation of test specimens and determination of properties'. Issued by the International Organization for Standardization (ISO).

ASTM D 1003 : 1992 'Test method for haze and luminous transmittance of transparent plastics'. Issued by the American Society for Testing and Materials (ASTM), USA

ASTM D 3935 : 1987 'Standard specification for polycarbonate (PC) unfilled and reinforced material'. Issued by the American Society for Testing and Materials (ASTM), USA

UL 94 : 1973 'Tests for flammability of plastic materials for parts in devices and appliances'. Issued by the Underwriters' Laboratories, Inc.

Indian Standard

POLYCARBONATE MOULDING AND EXTRUSION MATERIALS — SPECIFICATION

1 SCOPE

1.1 This standard covers the requirements, methods of sampling and tests for both polycarbonate homo-polymers and co-polymers, either filled or unfilled, suitably compounded to ensure desired performance of the parts manufactured from it by any of the usual processing techniques like injection moulding, extrusion, blow moulding, compression moulding etc.

1.2 The standard establishes a system for designating various possible polycarbonate materials. Since the system is not based on application, part design and performance requirements, it cannot be used for selection of a polycarbonate material for specific use. For selection of material for specific use, experts well versed with the application, part design, performance requirements, materials and processing techniques should be consulted. Similarly the code of good processing practices covered in this standard is intended to act as a guide to the processing community. For specific application and material, expert opinion should be sought for part and mould design, machine selection and processing parameters.

1.3 The classification system for various types of polycarbonate materials is based on levels of certain important physical, mechanical and thermal properties like:

- Melt flow
- Specific gravity
- Flexural modulus
- Impact strength
- Deflection temperature under load

and level and type of filler, presence of certain specific additives, colours, any specific feature of the material, processing technique and some specific performance requirements.

2 NORMATIVE REFERENCES

The following standards contain provision which through reference in the text, constitute provisions of this standard. At the time of publication the editions indicated were valid All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
3396 : 1979	Methods of tests for volume and surface resistivities of electrical insulating material (*first revision*)
4486 : 1967	Recommended methods for the determination of the permitivity and dielectric dissipation factor of electrical insulating materials at power, audio and radio frequencies including metre wavelengths
4905 : 1968	Methods for random sampling
8543 (Part 3/Sec 2): 1978	Methods of testing plastics: Part 3 Preparation of test specimens, Section 2 Injection moulded test specimens
13360 (Part 3/Sec 1): 1995 ISO 1183:1987	Plastics — Methods of testing: Part 3 Physical and dimensional properties, Section 1 Density and relative density of non-cellular plastics
13360 (Part 4/Sec 1): 1995 ISO 1133:1991	Plastics — Methods of testing: Part 4 Rheological properties, Section 1 Melt man-flow rate (MFR) and the melt volume flow rate (MVR) of thermoplastics
13411 : 1992	Glass reinforced polyester dough moulding compounds (DMC)

3 DESIGNATION/CLASSIFICATION SYSTEM

3.1 This standard adopts a data block system consisting of five blocks — each block describing specific information about the material. Each block is separated from the other by an asteric mark. In case a block is not used, the skipped block shall be indicated by an additional asteric mark.

Block 1 * Block 2 * Block 3 * Block 4 * Block 5

3.2 Information given in individual blocks shall be as follows:

3.2.1 *Block* 1

Contains this IS Specification number to indicate that the classification is according to this standard.

3.2.2 *Block* 2

This block is used to describe the thermoplastic material under consideration. Four letters and two digits are used for this purpose. First two letters 'PC' indicate that the material under designation is polycarbonate. Following two letters indicate the type and form of reinforcement or filler present and last two digits are for level of addition of the reinforcement or filler. The codes regarding the reinforcement/filler and the level of addition are given in Tables 1 and 2 respectively. In case of unfilled material the two letters and two digits succeeding 'PC' shall be expressed by use of four "X"s.

Example:

For a 20 percent Glass fibre filled polycarbonate material Block 2 shall be represented by PCGF20. Percentage of any inorganic filler/reiforcement shall be derived by method described in Annex B of this standard at temperature of 800 to 850°C.

Table 1 Codes for Filler/Reinforcement

(*Clause* 3.2.2)

Code (1)	Material (Position 3) (2)	Code (3)	Form (Position 4) (4)
C	Carbon	B	Ball/Beads/Spheres
G	Glass	P	Powder
T[1]	Mineral	F	Fibre
M[1]	Metal	G	Ground
O	Others	W	Whisker
X	Not specified	S	Scale/Flakes
		O	Others
		X	Not specified

[1] The material can be specified after the full designation of polycarbonat.e by chemical name or symbol.

3.2.3 *Block* 3

This block accommodates four letters. The first letter always indicates the processing technique for which the material is suitable. The other three letters indicate presence of specific additives, colours and any specific feature of the material. If any of the information is not relevant in this block, the same shall be represented by a "X". The codes for processing techniques and for additives and feature are given in Table 3.

Table 2 Codes for Percentage Content of Filler/Reinforcement

(*Clause* 3.2.2)

Code (1)	Percent, Content by Mass (Position 5 and 6) (2)
05	< 7.5
10	> 7.5 to 12.5
15	> 12.5 to 17.5
20	> 17.5 to 22.5
25	> 22.5 to 27.5
30	> 27.5 to 32.5
35	> 32.5 to 37.5
40	> 37.5 to 42.5
45	> 42.5 to 47.5
50	> 47.5 to 55
60	> 55.0 to 65
70	> 65.0 to 75
80	> 75.0 to 85
90	> 85.0

Table 3 Codes for Processing Techniques and Additives

(*Clause* 3.2.3)

Code (1)	Position (2)	Code (3)	Position 2 to 4 (4)
B	Blow moulding	C	Coloured
E	Extrusion	E	Expandable
I	Injection moulding	F	Flame retardant
C	Compression moulding	H	Heat stabilized
O	Optical disc moulding	L	Light and/or weather stabilized
R	Rotational moulding	M	Food and medical approved
G	General purpose	N	Natural (not coloured)
X	No indication	P	Impact modified
		R	Mould release agent
		T	Improved transparency
		W	Stabilized against hydrolysis
		Y	Increased electrical conductivity
		Z	Antistatic

Example:

A general purpose light stablized polycarbonate material containing mould release additives and in natural colour shall be represented in Block 3 as GLRN.

3.2.4 *Block* 4

Combination of five digits form this block. Each digit indicates the following properties in order:

i) Melt flow index at 300°C under 1.2 kg load as per IS 13360 (Part 4/Sec 1) /ISO 1133 when measured after predrying of the material at 120 ± 5°C (effective) for not less than 6 h. Codes for various levels of melt flow index are given in Table 4.

ii) Specific gravity of the material when measured as per IS 13360 (Part 3/Sec 1) / ISO 1183. Codes are given in Table 4.

iii) Flexural modulous of the material when measured as per Annex F of IS 13411 with crosshead speed of 1.3 mm/min and a span to depth ratio of 16 to 1. Test specimen used should be 3.2 mm in depth and 12.7 mm in width. Codes are given in Table 4.

iv) Izod impact strength, notched, when measured as per Annex E of IS 13411 with test specimen thickness of 3.2 mm and notch radius of 0.25 mm. Codes are given in Table 4.

v) Deflection temperature under load of 1.82 MPa when tested as per Annex H of IS 13411 using a 3.2 mm thick test specimen. Codes are given in Table 4.

If any of the above properties are not to be specified or not available a "X" mark shall be included in the block at appropriate position.

Example:

For a polycarbonate material with MFI between 3 to 6, specific gravity of 1.25, flexural modulous of 3 700 MPa, izod impact strength, notched of 250J/m and deflection temperature under load of 143°C, Block 4 will look like 22265.

3.2.5 *Block* 5

This block is provided for any additional specific performance requirement if required to be specified. These specific performances include electrical, flammability and optical requirements. In case, there is no specific requirement for the material the designation ends at Block 4 with an asterisk mark.

Each requirement is codified by a combination of one letter and two digits. The letter indicates the requirement; the subsequent digit indicates the thickness of the test specimen and the last digit indicates the specified properties. The scheme is elaborated below:

E = Electrical requirement

First digit

0 = Specimen thickness to be decided mutually by user and supplier.

1 = Specimen thickness 3.2 mm nominal

Table 4 Codes for Block 4

(*Clause* 3.2.4)

Code	Melt Flow Index g/10 min, 300°C, 1.2 kg Load	Code	Specific Gravity	Code	Flexural Modulus MPa, *Min*	Code	Izod Impact Strength, Notched, J/m, *Min*	Code	Deflection Temperature Under 1.82 MPa Load, °C, *Min*
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)
1	< 3	1	1.19 to 1.22	1	2 000	1	700	1	120
2	> 3 to 6	2	>1.22 to 1.3	2	3 000	2	600	2	125
3	> 6 to 12	3	>1.3 to 1.38	3	4 500	3	500	3	130
4	>12 to 24	4	>1.38 to 1.50	4	6 000	4	400	4	135
5	>24	5	>1.50	5	7 500	5	300	5	140
				6	9 000	6	200	6	145
				7	10 500	7	100	7	150
				8	12 000			8	155
X	No indication	X	No indication	X	No indication	X	No indication	X	No indication

Second digit

0 = Properties to be decided mutually by user and supplier

1 = Electrical properties conform with property limit mentioned in Table 5

2 = Electrical properties conform with property limit mentioned in Table 6

NOTE — Though the above system is infinitely flexible and can be used theoretically to express any desired combination, the user should keep in mind that certain combinations are not practical and the buyer and seller should mutually agree before specifying any unusual combination.

F = Flammability requirements in accordance with Annex C of this standard (as per UL 94)

First digit

0 = Specimen thickness to be decided mutually by user and supplier

1 = Test specimen thickness 6.10 mm maximum

2 = Test specimen thickness 3.05 mm maximum

3 = Test specimen thickness 1.47 mm maximum

Second digit

0 = Flammability class to be decided mutually by the user and supplier

1 = UL 94 HB Flammability class

2 = UL 94 V2 Flammability class

3 = UL 94 V1 Flammability class

4 = UL 94 V0 Flammability class

5 = UL 94 5V Flammability class

NOTE — This does not intend that above flammability rating by any way reflects hazards presented under actual fire conditions.

T = Optical requirements in accordance with Annex D of this standard

First digit

0 = Specimen thickness to be mutually decided by user and supplier

1 = Specimen thickness 6.4 mm nominal

2 = Specimen thickness 3.2 mm nominal

3 = Specimen thickness 1.6 mm nominal

Second digit

0 = Percentage transmittance to be mutually decided by user and supplier

1 = Minimum 88 percent transmittance

2 = Minimum 85 percent transmittance

3 = Minimum 82 percent transmittance

Table 5 Electrical Properties of Unreinforced Polycarbonate Materials

(*Clause* 3.2.5)

Property (1)	Test Method, Ref to IS (2)	Requirement (3)
Volume resistivity, *Min.* ohm-cm	3396	1×10^{15}
Dielectric strength, *Min.* kV/mm	4486	14
Dielectric constant at 1 MHz, *Max*	do	3.0
Dissipation factor at 1 MHz, *Max*	do	0.01

Table 6 Electrical Properties of Reinforced Polycarbonate Materials

(*Clause* 3.2.5)

Property (1)	Test Method, Ref to IS (2)	Requirement (3)
Volume resistivity, *Min.* ohm-cm	3396	1×10^{14}
Dielectric strength, *Min.* kV/mm	4486	15
Dielectric constant at 1 MHz, *Max*	do	3.7
Dissipation factor at 1 MHz, *Max*	do	0.015

Example 1

IS **PCGF10** **ILRC** **22265** **F34**

Block 1
(Indian Standard)

Block 2
[Glass fibre filled (between 7.5 to 12.5 percent) polycarbonate]

Block 3
(Suitable for injection moulding, light stabilized contains mould release agent and colours)

Block 4
(MFI between 3 to 6, specific gravity between 1.22 to 1.3, flexural modulus of 3 000 MPa, *Min*; Izod impact strength-notched of 200 J/m, *Min* and DTUL of 140 °C, *Min*)

Block 5
(Meets UL 94 V-0 flammability class at 1.47 *Max* sample thickness)

Example 2

IS **PC XXXX** **BMWN** **21124**

Block 1
(Indian Standard)

Block 2
(Unfilled polycarbonate material)

Block 3
(Suitable for blow moulding, food and medical approved, stabilized against hydrolysis and natural in colour)

Block 4
(MFI between 3 to 6, specific gravity between 1.19 to 1.22, flexural modulus of 2 000 MPa, *Min*; Izod impact strength-notched of 600 J/m, *Min* and DTUL of 135°C, *Min*

4 CONFORMANCE TO THE STANDARD

4.1 All property limits specified in this standard are considered to be absolute in nature.

4.2 To check conformity of any material with this standard, observed or calculated values of properties have to be directly compared with the specified limits without taking recourse to rounding off.

4.3 Conformance or non-conformance is decided based on this comparison.

5 SAMPLING

Unless otherwise agreed upon between the user and the supplier, the materials shall be sampled in accordance with Annex E. Adequate statistical sampling shall be considered as an acceptable alternative. A lot of resin shall be considered as a unit of manufacture as prepared for shipment, and may consist of a blend of low or more production runs or batches of material.

6 PREPARATION OF TEST SPECIMENS

Test specimens for properties mentioned in (iii) to (v) of **3.2.4** are to be made in accordance with IS 8543 (Part 3/Sec 2).

7 CONDITIONING OF TEST SPECIMEN AND CONDITIONS FOR TESTS

All test specimen are to be conditioned for a minimum period of 48 h at 27 ± 2°C under 65 ± 5 percent relative humidity and the tests are also to be carried under same conditions.

8 PACKAGING AND MARKING

8.1 Packing

Packaging should usually be the standard packaging adopted by the supplier unless otherwise agreed with the user. Packaging must ensure adequate protection of the content from contamination and loss of material. Mass of the packages should usually be 25 kg nominal unless otherwise agreed between the user and the supplier.

8.2 Marking

Each packages shall be securely closed and legibly and indelibly marked with the following information:

i) Indication of the source of manufacture and recognized trade-mark, if any;
ii) Name and designation of the material;
iii) Net mass;
iv) Date of manufacture; and
v) Batch No. or Code No.

8.3 BIS Certification Marking

The packages may also be marked with the Standard Mark

8.3.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturer or producers, may be obtained from the Bureau of Indian Standards.

ANNEX A

(*Foreword*)

CODE OF GOOD PROCESSING PRACTICES

A-1 Polycarbonate is a relatively new material to the Indian plastic processing community and user industries in general. Improper processing and design considerations many a times lead to disasterous results which are not expected from polycarbonate. This code of good processing practices highlights the areas of critical importance in processing and tool design without any pretension of a full scale guide to moulding of polycarbonate. It is strongly advised that experts especially the raw material supplier should always be consulted for every aspect of product design, tool design and finally processing of polycarbonate.

A-2 PRE-DRYING

A-2.1 Polycarbonate materials do not absorb much moisture. But presence of moisture beyond 0.02 percent level in polycarbonate is enough to cause hydrolytic polymer chain degradation at processing temperature resulting in highly brittle product. To avoid this eventuality, it is essential to pre-dry any polycarbonate material effectively at 120 ± 5°C for at least 4 h. Effect of pre-drying time at 120 ± 5°C on moisture content and impact strength is depicted in Fig. 1 and Fig. 2 respectively.

A-2.2 Pre-drying of polycarbonate granules can be carried out in any of the following type of dryers:

- Air circulating oven
- Forced air circulating dryer
- Dry air circulating dryer

In the first case the depth of granules on the tray should not exceed 2.5 cm. Enough space should be maintained between trays to allow adequate air circulation. In any of the above drying arrangement cleanliness is very important to avoid unwarranted material contamination.

A-2.3 Effectiveness of pre-drying on moisture content can be checked by a very easy test commonly known as 'Thomasetti's Volatile Indicator' (TVI). In this test two microscope slides are placed on a hot plate and allowed to be heated up to 290°C (the temperature is checked with a surface pyrometer). At least four granules from dryer are placed on the slide keeping some distance between each. Immediately the granules are covered with the other slide and the slides are pressed together with a straight edge till the granules are compressed to about 12mm diameter. Moisture is indicated by the presence of bubbles in the flattened granules. A few small bubbles denote moisture content of between 0.02 percent to 0.03 percent, numerous bubbles between 0.05 percent to 0.1 percent and many large bubbles above 0.1 percent. It should be noted that presence of one or two small bubbles in only one or two granules indicate trapped air rather than presence of moisture.

A-3 PROCESSING

A-3.1 Polycarbonate can be processed into useful end products by any of the usual processing techniques like extrusion, blow moulding, injection moulding etc. Selection of processing equipment, processing conditions and tool designing plays extremely important part in achieving best possible performance in end products made out of polycarbonate.

A-3.2 Selection of Equipment

A-3.2.1 *Extruder*

Extruded polycarbonate sheets and profiles find variety of applications. Use of twin screws extruder is to be avoided. Detailed geometry of single screw extruders without and with venting that should be selected for processing of polycarbonate is given in Table 7.

Table 7 Geometry of Single Screw Extruders

	3-Zone Unvented (1)	6-Zone Vented (2)
Feeding	6 - 8 D	5 - 7 D
Compression	4 - 5 D	4 - 5 D
Metering	11 - 13 D	4 - 5 D
L/D ratio	21 - 26 D	–
Compression ratio	1:2.5 - 2.8	1:2.5 - 2.8
Venting	—	4D
Compression	—	3 - 4 D
Metering	—	8 - 10 D
L/D ratio	—	28 - 35 D
Compression ratio	—	1:2.1 - 2.3

Grooved barrel improves better material transportation but care should be taken so that high shear does not lead to material overheating. Hopper block should preferably be sealed to prevent moisture pick up. To control feed and avoid premature melting, water cooling should be provided in the hopper block.

Heating of extruder barrel should be achieved by resistance heater band. Control system should be able to maintain close tolerance of ± 2 °C up to at least 325 °C. Thermostatically controlled air blower and deep probe thermocouples should be used for this purpose.

Like for any other plastic material, breaker plate/filter assembly has to be used for polycarbonate extrusion also to achieve better compression, melting and homogenization. This helps in eliminating contamination and reducing fish eyes. Filter combination of 40-60/100-120/40-60 mesh is preferred.

Drive power requirement for single screw extruder suitable for polycarbonate extrusion is usually in the range of 0.2 to 0.3 kWh/kg output as it depends on

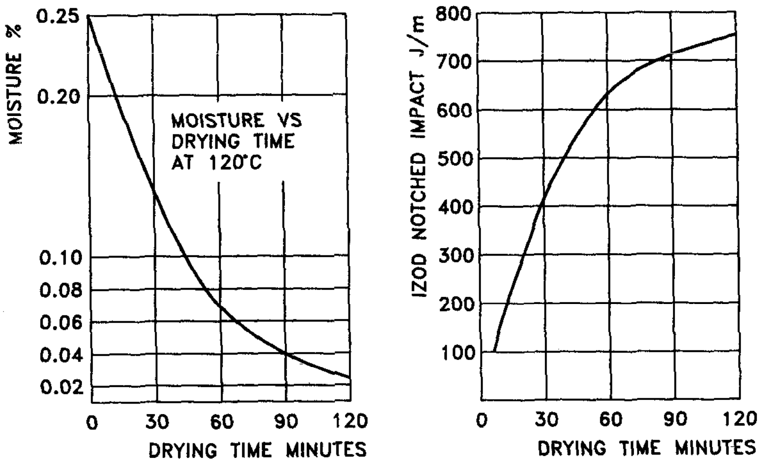


FIG. 1 AND FIG. 2 EFFECT OF DRYING ON POLYCARBONATE

melt temperature, viscosity and screw geometry. Power required for polycarbonate extrusion in single screw extruder with different screw diameters is given in Table 8.

Table 8 Power Requirement

Screw Diameter mm (1)	Required Power kW (2)
45	20-25
60	25-30
90	50-60
120	75-110

An ideal polycarbonate extruder should be equipped with an infinitely variable gear box to allow screw speed adjustment as a calibrating parameter.

A-3.2.2 *Extrusion Blow Moulding*

Continuous and intermittent parison extrusion, with or without accumulators, on both single and dual station machines can be used for blow moulding of polycarbonate. To achieve better resin stability, it is advisable to use chrome plated steel barrel instead of nitrided steel. Minimum two heating zones are recommended for shorter barrels while at least three with separate heater bands for connecting flanges are to be used for larger barrels.

High compression mixing screws are to be completely avoided. For good results screws with compression ratio of 2:1 to 3:1 should be used. The minimum recommended L/D ratio of the extruder screw is 15:1.

All heating zones should be precisely controlled and no unheated gap should exist on the barrel. On-off pyrometer control should be avoided in favour of variable temperature regulator to achieve uniform melt.

Power requirement for the extruder in the blow moulding machine suitable for polycarbonate is in the range of 0.2 to 0.3 kWh for every kg/h of resin. A screw torque of at least 2 700 kg.cm up to 3 600 kg.cm for low screw rpm should be achieved for better results. A typical 21:1, L/D ratio screw in the extruder section of a blow moulding machine for polycarbonate should have its zones divided in 7D feed, 9D transition and 5D metering for best results.

A-3.2.3 *Injection Moulding*

To injection mould polycarbonate, plunger type machines are positively to be avoided. Screw pre-plasticising type injection moulding machines preferably with medium to high injection pressure capability usually give satisfactory result. Machines equipped with precise temperature pressure and speed control give optimum productivity and quality parts.

Hourly plasticising capacity of the machine should be adequate to take care of total part weight produced during the hour. Too high a capacity may lead to poor strength in the part due to thermal degradation resulted from high residual time of the melt in the barrel. Lower capacity will extend the cycle time, thereby reducing the productivity.

Shot capacity of the injection moulding machine should be at least 20 to 30 percent more than the part weight including sprue and runners to avoid incomplete parts. Part weight should also not be lower than 20 percent of the shot capacity to avoid thermal degradation of the melt remaining in the heated machine barrel for longer period of time. The clamping force of the machine is usually decided on the basis of projected area of the part and injection pressure which in turn depends on part intricacies, flow length and thickness of the part. To take an example, a part with 400 cm^2 projected area being moulded at an injection pressure of 1 000 kg/cm^2 will need a machine with minimum clamping force of 200 tons (it is assumed that only 50 percent of injection pressure is actually transmitted into the mould cavity due to pressure drop in flow path).

L/D ratio of the plasticising screw of an injection moulding machine processing polycarbonate should be 20:1 and the compression ratio should be in the range of 1:2 to 1:2.5. Shut-off valves cause material hang over and consequent thermal degradation resulting in poor quality parts. Short free flow nozzles with a minimum opening of 3mm are recommended for processing of polycarbonate. Good nozzle temperature control improves part quality and eliminates melt drooling.

Since polycarbonate is used in components of precision engineering applications, dimensional accuracy and high performance levels have to be assured during production of the parts. Parameters like temperature, pressure and speed influence part quality immensely and they are to be precisely controlled to ensure quality parts. The machine should be able to control the temperature within ± 2 °C of the settings. Modern injection moulding machines are equipped with microprocessor control and various control options like variable pressure and speed control, cavity pressure regulation, time control, viscosity regulator etc are available. These are extremely useful in achieving superior part quality and should be preferred whenever possible.

A-3.3 Dies and Moulds

A-3.3.1 *Extrusion Dies*

Extrusion die must ensure smooth flow of the melt. All possible areas of material hang up must be avoided. To ensure uniform wall thickness of the extruded profile dies with adjustable opening is preferred. In bigger dies segmented temperature control arrangements help in achieving superior quality product. Downstream equipments in an extrusion line will depend on the profile to be extruded. Equipment supplier and experts are to be consulted in designing the downstream.

A-3.3.2 *Blow Moulding Dies and Mould*

Integrated head which combines the function of head and accumulator in a single unit is preferred for extrusion blow moulding of polycarbonate. Uniform heat distribution and wall thickness control are ensured in this system.

Streamlined dies ensure smooth flow of the melt without any possibility of hang up. To achieve better transparency in the product highly polished die and head should be used.

Die should be equipped with adjustable bushing to achieve parison thickness uniformity. As polycarbonate does not have appreciable die swell characteristics, the die gap should be very close to the final wall thickness of the parison. The die bushing and mandrel should be maintained at the same level. Radii at the orifice should be around 0.5 mm. Die bushing and mandrel should be equipped with separate temperature control to prevent rolling of parison. Die and length should be 10 to 15 times the parison slot width, but generally not exceeding 10mm. Dark red hot wire cutter or conventional water cooled thin cutting blade can be used. For strong weld lines, the pinch-off blades should have included angle of 30-45 degrees and the blade width should be maintained between 0.05 - 0.08 mm (*see* Fig.3).

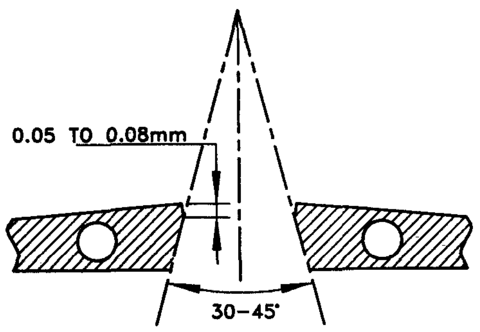


FIG. 3 BLOW MOULD PINCH OFF BLADES

Satisfactory result could be achieved by use of alluminium or berryllium copper alloys for mould construction. However, if very high part quality is desired, steel is the preferred material. Heating and cooling channel should be provided to help speed of production and quality of parts. To eliminate possibility of stress concentration in the part, sharp corners should be avoided in the mould. Minimum radii of 0.25 mm must be incorporated at all corners. Pinch off areas should be generous and deep under cuts should be avoided. Finely roughened mould surface aids in avoiding air entrapment. To achieve maximum pressure at the parting line the mould faces should be relieved.

A-3.3.3 *Injection Moulds*

Sprue bushing in an injection mould should have a generous taper, should be well polished and hardened. The sprue diameter should be larger than the nozzle orifice by 1 mm. The sprue bushing radius should be 1 mm larger than that of the nozzle. The small end of the sprue should be at least 4 mm in diameter. In case of direct sprue gating, cooling opposite the sprue avoids sink marks and cooling in the sprue area reduces cycle time.

Short full round primary runners with at least 5 mm diameter are preferred. For long flow path hot runners are recommended. Cold slug wells at the end of both primary and secondary runners must be provided. Trapezoidal runners can be used if necessary, but half round runners should be avoided.

Gate type, dimension and location depends on the product design and on polycarbonate grade like unreinforced and reinforced and its viscosity. All types of gates, namely pin point, diaphragm, film, tunnel, fan, tab, spider, ring, etc could be successfully used for polycarbonate. For unreinforced polycarbonate the pin point gate diameter should be between 0.8 to 2 mm while the same for reinforced material should be between 1 to 2.5 mm. The gate should have very short or no land length. A maximum land length of 0.5 to 1 mm is recommended in case of diaphragm gating. In case of tunnel gate, the minimum diameter for unreinforced polycarbonate should be 0.8 mm and the same should be minimum 2 mm for reinforced material. In deciding the location of the gate following points should be kept in mind:

i) Gate should be placed preferable in the thickest section of the part,

ii) Gate location should minimize weld lines,

iii) Gate should direct air towards the vents, and

iv) Location should facilitate degating.

Like the sprue, runner and gating the location and design of venting is also important. Improper venting could cause air entrapment in the mould leading to localized burning, sinks, short shots, high moulded-in stress etc. For shallow articles 0.02 to 0.05 mm thick vents should be placed at intervals of 2 to 4 cm around to cavity periphery which allows venting of air on the split lines opposite the gate. Too much of venting, however, can cause reduction of air speed and can cause blockage and it can also act as potential area for flash. For more complicated and large products venting should be decided on the basis of product and mould design.

To eliminate stress concentration all sharp corners are to be avoided and radii are to be incorporated. For easy removal of part from mould, a draft of approximately 1/2° to 2° per side for both internal and external walls is recommended. In case of complex parts higher draft might be required. In case of textured finish, draft angles should be increased by at least 1° per side for every 0.025 mm of texture depth.

A-3.4 Processing Condition

A-3.4.1 *Extrusion*

While extruding flat sheets, depending on the grades of polycarbonate used, the melt temperature should usually be maintained around 250 °C. Temperature of various zones, die and orifice are adjusted accordingly. Die and orifice temperature should be maintained 5 to 10 °C lower to the adopter temperature. Calender rolls should be placed as close to the die as possible. Temperature of the rolls should be between about 130 to 160 °C. In case of vented barrel extruders which give better extrusion results, however, temperature setting are different and has to be decided based on experience with a particular extruder. In case of other profiles the conditions depend on grade used and intricacy of the profiles. In polycarbonate extrusion oil coolded screw gives better result especially in terms of elimination of surging.

A-3.4.2 *Blow Moulding*

To start the machine the temperatures should be to 10 °C higher than the normal operational level for polycarbonate. This prevents possible damage to the equipment resulting from high back pressure generated through cold melt. Depending on the grade used the melt temperature should be around 260 °C. Mould temperature of about 60-80 °C gives good transparency and surface finish as well as shorter cycle time. Since polycarbonate melt does not have the desired elasticity, parison formation and blowing should be accomplished as rapidly as possible. Top blowing is widely used for products up to 2 l. For larger products, bottom blowing is preferred. Air pressure for blowing should be maintained between 1.5 to 3.5 kgf/cm^2. Blow ratios of up to 4:1 can be adopted without much problem.

A-3.4.3 *Injection Moulding*

The product, mould design and the grade used decide the temperature condition in injection moulding of polycarbonate. Depending on grades used melt temperature between 280 to 320 °C has to be achieved to ensure easy moulding and quality product. Mould temperature between 80-120 °C not only helps in easy flow of melt in the mould and shorter cycle time, it also ensures stress free moulding with excellent surface finish.

Polycarbonate melt is usually highly viscous and injection pressure requirement is higher as compared to other thermoplastics. Depending on product size, gate dimension and grade used, quite often injection pressure higher than 1 000 kg/cm^2 is encountered. After or hold on pressure in case of polycarbonate moulding is about 50 percent of injection pressure. Back pressure should not exceed 10 kg/cm^2. Like other parameters, injection speed also depends on part intricacies, the mould quality and the gating system. Fast injection speed is, however, desirable to avoid premature freezing of the melt and knitting around corners. Slow injection speed should be used during start up. To facilitate release of part from mould, sometimes use of mould release agent like silicones spray or zinc stearate spray might be required. If, however, the parts need painting or decorating use of release agent should be avoided. These release agents should never be mixed with the polycarbonate granules.

A-4 OTHER ASPECTS

A-4.1 Shut Down and Purging

Polycarbonate should never be left in the barrel overnight or during weekends. However, for shorter shut down this can be allowed under following precautions:

- Barrel temperature is reduced to 170-180°C
- Heaters are kept on
- Temperature is never allowed to drop below 160°C

Below 160 °C polycarbonate tends to adhere to barrel wall and may pull off metal particles and degraded resin as it cools and contracts. Black specs may result in parts when production is restarted after such a temperature drop.

For longer shut down, the barrel should be emptied completely followed by a thorough purging using scrap acrylic or clear polystyrene. HDPE can also be used. LDPE should, however, be avoided.

A-4.2 Recycling

Up to 20 percent of first generation scrap generated in-house the processing shop can be blended with virgin polycarbonate for processing. However, the cleanliness of the scrap is of utmost importance to achieve quality production. Proper pre-drying of the reground scrap as described for virgin material is also absolutely essential for recycling purpose.

A-4.3 Quality Control of Moulded Parts in Polycarbonate by Means of Stress Corrosive Liquids

The aim of quality control of moulded parts should be a reproducible indication for moulder and end user that processing conditions are such that parts of sufficient quality and with required properties are produced.

Causes of an early breakage of a part may include the presence of excessive stresses, due to wrong processing leading to moulded-in stresses, and assembly stresses.

Generally these stresses cannot be predicted nor calculated, but can be estimated by means of immersion of the moulding in a stress corrosive liquid. These liquids are carefully selected for the different materials to show a stress cracking phenomenon under test conditions at stress levels around the maximum recommended working stresses.

Required liquids for polycarbonate are:

> Mixture of toluene and *n*-propanol (TnP) in mix ratios (vol) 1:10 and 1:3.

It should be emphasized, that these tests have to be carried out in carefully controlled test conditions to ensure reproducibility.

Furthermore, parts subjected to this test but not showing crazes should be destroyed as well, their expected life time has been drastically reduced.

All these tests are destructive they have to be carried out as spot checks in a statistically justified manner.

Care should be taken not to pollute the liquids with other solvents, which may cause stress cracking as well.

Liquids used daily should be replaced at least once every month.

This test is suitable for all unreinforced regular grades of polycarbonate. A mixture of toluene:*n*-propanol of 1:10 detects a stress level of approx. 20 N/mm^2. Below this level of polycarbonate part will generally have a sufficient lifetime. For more critical applications it is advisable to carry out the test with a mixture of toluene:*n*-propanol of 1:3 to detect stress levels higher than 10 N/mm^2.

When testing actual parts, the following procedure should be used:

i) Choose the appropriate mixture and allow it to come to room temperature.

ii) Immerse the polycarbonate part in the bath for 3 min.

iii) Lift the part from the bath and rinse it with ethanol or propanol, dry it with compressed air and check it for cracks within 1 min after removal from the bath.

When no cracks are visible the stress level is below the above indicated levels.

NOTES

1 Other solvent or solvent mixtures are described for detecting stress levels in polycarbonate, for example tetrachloromethane and mixtures of ethyl-acetate and methanol. The tetra method is sensitive for stresses even lower than 3N/mm^2 and therefore leads to an unnecessarily severe judgement.

Main drawbacks of the ethyl-acetate/methanol mixtures are that ethyl-acetate if absorbed by polycarbonate, so the test solvent should only be used once. Furthermore, the method is very sensitive to temperature and mix-ratio.

2 TnP mixtures should be stored in well closed flasks and containers in order to keep the mix ratio constant.

ANNEX B

(*Clause* 3.2.2)

DETERMINATION OF ASH

B-1 PRINCIPLE

It is possible to determine the ash of an organic material by three main methods:

a) Direct calcination, that is by burning the organic matter and treating the residue at high temperature until constant mass is reached (Method A).

b) Calcination after sulphation, which may be carried out by two different procedures:

- With sulphuric acid treatment after burning, that is by burning the organic matter transforming the inorganic residue into sulphates with concentrated sulphuric acid and treating the residue at high temperature until constant mass is reached. This is the common method of obtaining 'sulphated ash' (Method B).
- With sulphuric acid treatment before burning, that is by heating the organic matter together with concentrated sulphuric acid up to temperatures where fuming and subsequent burning of the organic matter occur, and finally treating the residue at high temperature until constant mass is reached. This procedure may be applied if volatile metal halides are liable to evaporate during burning of the organic matter. It should never be applied to silicones or fluorine-containing polymers (Method C).

In each case the final step of the operation is a calcination at 600 °C, 750 °C, 850 °C or 950 °C, until constant mass is reached (*see* **B-4.2**).

B-2 REAGENTS (for methods B and C only)

During the analysis, use only reagents of analytical grade and only distilled water or water of equivalent purity.

B-2.1 Ammonium Carbonate, Anhydrous

B-2.2 Ammonium Nitrate, approximately 10 percent (*m/m*) solution.

B-2.3 Sulphuric Acid, 1.84 g/ml.

B-2.4 Sulphuric Acid, 50 percent (*v/v*) solution.

B-3 APPARATUS

B-3.1 Crucible of silica, porcelain or platinum, inert to the material tested.

B-3.2 Gas Burner, or other appropriate heat source.

B-3.3 Muffle Furnace, capable of being controlled at 600 ± 25 °C, 750 ± 50 °C, 805 ± 50 °C or 950 ± 50 °C as appropriate.

B-3.4 Analytical Balance, accurate to 0.1 mg.

B-3.5 Pipettes, of suitable capacity (for methods B and C only).

B-3.6 Desiccator, containing an efficient desiccant which does not interact with the ash.

NOTE — In certain cases, the ash may have a greater affinity for water than some substances commonly used as desiccants.

B-3.7 Weighing Bottle

B-4 PROCEDURE

B-4.1 Test Portion

Take a quantity of the test sample sufficient to yield 5 to 50 mg of ash. If the approximate ash is unknown, carry out a preliminary ash determination.

NOTE — According to the result obtained, the recommended test portion is chosen from the Table 9.

Table 9 Recommended Test Portion

(*Clause* 4.1)

Approximate Ash percent (1)	Test Portion g (2)	Mass of Ash Obtained mg (3)
< 0.01	200, *Min*	5 to 50
> 0.01 to 0.05	100	10 to 50
> 0.05 to 0.1	50	25 to 50
> 0.1 to 0.2	25	25 to 50
> 0.2	10, *Max*	20 to 50

For plastics yielding very low ash, it is necessary to use large test portions. When it is impossible to burn the whole of the test portion at one time, weigh the required quantity in a suitable weighing bottle and introduce it into the crucible in convenient amounts at a time for a succession of burnings until the whole of the test portion has been burnt.

B-4.2 Test Conditions

Calcination shall be continued to constant mass as defined in **B-4.3.6** but the duration of the calcination in the muffle furnace shall not exceed 3 h at the specified temperature.

The choice of the calcination temperature and the use of the sulphation method depend on the nature of the plastic and any additives it may contain. If a choice exists between different satisfactory conditions, choose those which allow the attainment of constant mass in less than 3 h. A higher temperature or the use of sulphation generally shortens the duration of the calcination.

Whichever method — A, B or C — is used, choose one of the following temperature ranges for the final step of calcination, unless other temperatures are requested for special technical or commercial reasons:

600 ± 25 °C, 750 ± 50 °C, 850 ± 50 °C,
950 ± 50 °C

B-4.3 Method A — Direct Calcination

B-4.3.1 Prepare the crucible by heating it in the muffle furnace at the test temperature until constant mass is reached. Allow to cool in the desiccator to room temperature for at least 1 h and weigh on the analytical balance to the nearest 0.1 mg.

B-4.3.2 Introduce into the tared weighing bottle the test portion, pre-dried as described in the corresponding material specification or with a known volatile matter content corresponding to 5 to 50 mg of ash. Weigh again to the nearest 0.1 mg or to 0.1 percent of the mass of the test portion. If the crucible will accommodate the test portion corresponding to the 5 to 50 mg of ash, this quantity may be placed directly into the crucible and weighed in it. High bulk materials may be compressed into tablets which may then be broken up into fragments of appropriate size.

B-4.3.3 Introduce into the crucible enough of the material to be calcined so that it is not more than half full. Heat the crucible directly on the burner or other suitable heating device to burn slowly. Burning shall not be too vigorous to avoid loss of ash particles. Cool and add another part of the test portion. Repeat the operation described until the whole test portion is consumed.

B-4.3.4 Introduce the crucible into the muffle furnace preheated to the prescribed temperature and calcine for 30 min.

B-4.3.5 Place the crucible in the desiccator, allow it to cool to room temperature for 1h and weigh on the analytical balance to the nearest 0.1 mg.

B-4.3.6 Calcine again under the same conditions until constant mass is reached, that is, until the results of two consecutive weighings do not differ from each other by more than 0.5 mg.

B-4.4 Method B — Calcination Following Sulphuric Acid Treatment After Burning

B-4.4.1 Proceed as specified in **B-4.3.1**, **B-4.3.2** and **B-4.3.3**.

B-4.4.2 After cooling, add the sulphuric acid solution drop by drop with a pipette of suitable capacity to moisten the residue completely and heat until fuming ceases, avoiding too vigorous boiling.

B-4.4.3 If traces of carbonaceous materials remain after cooling, add 1 to 5 drops of the ammonium nitrate solution and heat until the evolution of white fumes ceases completely.

B-4.4.4 In order to reconvert metal oxides formed during the preceding steps into sulphates, add, after cooling, about 5 drops of the concentrated sulphuric acid and heat until there is no further evolution of white fumes, avoiding vigorous boiling or the loss of ash by excessive fuming.

B-4.4.5 After cooling, add 1 to 2 g of the solid ammonium carbonate and heat, avoiding loss of ash, until the fuming has ceased. Then place the crucible in the muffle furnace preheated to the indicated temperature and proceed as specified in **B-4.3.4**, **B-4.3.5** and **B-4.3.6**.

B-4.5 Method C — Calcination Following Sulphuric Acid Treatment Before Burning

B-4.5.1 Proceed as specified in **B-4.3.1** and **B-4.3.2**.

B-4.5.2 Introduce into the crucible enough of the material to be calcined so that it is not more than half full. Add with a pipette a sufficient amount of the concentrated sulphuric acid to moisten the material completely. Cover the crucible with a watch-glass. Heat the crucible directly on the burner at low flame until the organic material begins to decompose.

Continue heating carefully, adjusting the watch-glass so as to allow the acid to be fumed off, making sure that no ash-containing material is lost. With plastics which have a tendency to lose ash-containing material, it is recommended that the crucible with its contents be placed into a holed asbestos board and heated with a low flame only until the organic matter smoulders rather than burns. If the initial charge into the crucible was insufficient to yield an acceptable mass of ash, allow the crucible to cool, add another part of the test portion and repeat the operation described before until the whole test portion is burned in the crucible. Remove the watch-glass, making sure that no solid particles are adhering to it.

NOTE — In cases where the sulphuric acid tends to creep over the lip of the crucible or where, despite precautions, some of the test portion tends to be lost from it by violent reaction (frequently in the case of PVC), the concentrated sulphuric acid may be replaced by a mixture of concentrated acetic and sulphuric acids. The use of these mixed acids should be agreed between the interested parties and reference made to it in the test report.

B-4.5.3 Proceed as specified in **B-4.4.3**, **B-4.4.4** and **B-4.4.5**.

B-5 NUMBER OF TESTS

The number of tests and the permissible scatter of results shall be stated in the particular standard for each material. If such information is not available, carry out two determinations and repeat the test as necessary until the results of two successive determinations do not differ from each other by more than 10 percent of their mean.

B-6 EXPRESSION OF RESULTS

The ash or sulphated ash expressed as a percentage by mass, is given by the formula

$$\frac{m_1}{m_2} \times 100$$

where

m_1 = is the mass, in g, of the test portion, and

m_2 = is the mass, in g, of ash obtained.

ANNEX C

(*Clause* 3.2.5)

TEST FOR FLAMMABILITY

C-1 GENERAL

C-1.1 These requirements cover tests for flammability of plastics materials used for parts in devices and appliances. They are intended to serve as a preliminary indication of their acceptability with respect to flammability for a particular application.

C-1.2 The methods described in this standard involve standard size specimens and are intended to be used solely to measure and describe the flammability properties of materials, used in devices and appliances, in response to heat and flame under controlled laboratory conditions. The actual response to heat and flame of materials depends upon the size and form, and also on the end-use of the product using the material. Assessment of other important characteristics in the end-use application includes, but is not limited to, factors such as ease of ignition, burning rate, flame spread, fuel contribution, intensity of burning, and products of combustion.

C-1.3 The final acceptance of the material is dependent upon its use in complete equipment that conforms with the standard applicable to such equipment. The flammability classification considered for a material may vary, depending on the equipment or device involved and the particular use of the material. The performance level of a material by these methods should not be assumed to correlate with its performance in end-use application.

C-1.4 The requirements may be applied to other nonmetallic materials if found to be appropriate.

C-1.5 These requirements do not cover plastics when used as materials for building construction of finishing.

C-2 HORIZONTAL BURNING TEST FOR CLASSIFYING MATERIALS 94HB

C-2.1 Test Criteria

A material shall be classified 94HB on the basis of test results obtained on small bar specimens when tested as described in **C-2.4** to **C-2.12**.

C-2.2 A material classed 94HB shall (*see also* **C 2.3**):

i) not have a burning rate exceeding 38.1 mm per minute over a 76.2 mm span for specimens having thickness of 3.05-12.7 mm, or

ii) not have a burning rate exceeding 76.2 mm per minute over a 76.2 mm span for specimens having a thickness less than 3.05 mm, or

iii) cease to burn before the flame reaches the 102 mm reference mark (*see* **C-2.10**).

C-2.3 If only one specimen from a set of three specimens does not comply with the requirements, another set of three specimens is to be tested. All specimens from this second set shall comply with the requirements in order for material in that thickness to be classified 94HB.

C-2.4 Apparatus

The apparatus employed is to consist of the following:

a) *Test Chamber* — enclosure, or laboratory hood free of induced or forced draft during tests.

b) *Laboratory Burner* — A Bunsen or Tirrill burner having a tube with a length of 80-100 mm and an inside diameter of $9.4^{+1.6}_{-0.0}$ mm. The tube is not to be equipped with an end attachment, such as a stabilizer.

c) *Wire Gauze* — A 20 mesh (20 openings per 25.4 mm), 0.43 mm diameter iron wire gauze, 127 mm square.

d) *Gas Supply* — A supply of technical grade methane gas with regulator and meter for uniform gas flow. Natural gas having a heat content of approximately 37 MJ/m^3 has been found to provide similar results. Other fuel gases, such as butane, propane and acetylene (which have a higher heat value) may also be used. However, technical grade methane gas shall be used in case of dispute.

e) *Ring Stand* — A ring stand with clamps, or the equivalent, for horizontal positioning of the specimen and the wire gauze.

f) Stopwatch or other timing device.

g) Conditioning room or chamber capable of being maintained at 27 ± 2°C and a relative humidity of 65 ± 5 percent.

h) A metal support fixture as illustrated in Fig. 4 for testing specimens that sag or bend at their free end.

C-2.5 Test Specimens

Test specimens are to be limited to a maximum thickness of 12.7 mm width are to be provided in the minimum thickness and in a 3.18 ± 0.13 mm thickness. The 3.18 ± 0.13 mm thick specimens are not necessary if the minimum thickness is greater than 3.18 ± 0.13 mm or the maximum thickness is less than 3.18 ± 0.13 mm.

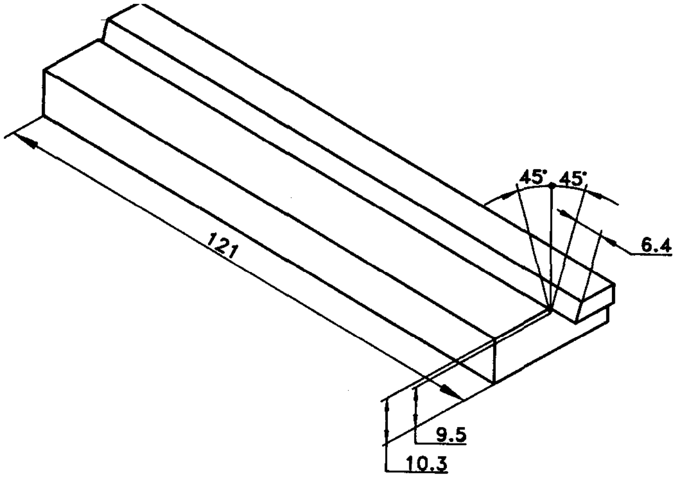
Exception: A material classified 94HB in the 3.18 ± 0.13 mm thickness shall automatically be classed 94 HB down to a 1.57 mm minimum thickness without additional testing.

C-2.6 The specimens are to comply with the following:

i) The maximum width is to be 213.2 mm.

ii) The edges are to be smooth, and the radius on the corners is not to exceed 1.3 mm.

C-2.7 If a material is to be considered in a range of colours, melt flows, or reinforcements, specimens



All dimensions in millimetres.

FIG. 4 FLEXIBLE SPECIMEN SUPPORT FIXTURE

representing these ranges are also to be provided. Specimens in the natural (if used in this colour) and in the most heavily pigmented light and dark colours are to be provided and considered representative of the colour range, if the test results are essentially the same. An additional set of specimens is to be provided in the heaviest organic pigment loading, unless the most heavily pigmented light and dark colours include the highest organic pigment level. When certain color pigments (for example, red, yellow, or the like) are known to have particularly critical effects, they are also to be provided. Specimens in the extremes of the melt flows and reinforcement contents are to be provided and considered representative of the range, if the test results are essentially the same. If the burning characteristics are not essentially the same for all specimens representing the range, evaluation is to be limited only to the material in the colours, melt flows, and reinforcement contents test, or additional specimens in intermediate colours, melt flows, and reinforcement contents are to be provided for tests.

C-2.8 Specimen Conditioning

The specimens are to be conditioned for at least 48 h at 27 ± 2°C and a relative humidity of 65 ± 5 percent prior to testing.

C-2.9 Test Method

The burning test is to be conducted in a chamber, enclosure, or laboratory hood free of induced or forced draft. An enclosed laboratory hood with a heat-resistant-glass window and an exhaust fan for removing the products of combustion after the test is recommended.

C-2.10 Each specimen is to be marked across its width with two lines, 25.4 and 102 mm from one end of the specimen. The specimen is to be clamped at the end farthest from the 25.4 mm mark, with its longitudinal axis horizontal and its transverse axis inclined 45°. The wire gauze is to be clamped horizontally beneath the specimen, with a distance of 9.5 mm between the lowest edge of the specimen and the gauze, and with the free end of the specimen even with the edge of the gauze (*see* Fig. 5).

C-2.10.1 If the specimen sags at its free end during the initial set up, the support fixture illustrated in Fig.4 is to be positioned under the specimen with the small extending portion of the support fixture 19 mm from the free end of the specimen. Enough clearance is to be provided at the clamped end of the specimen so that the support fixture can be freely moved sidewards. As the flame front progresses along the specimen, the support fixture is to be withdrawn at the same rate.

C-2.11 The burner is then to be placed remote from the specimen, ignited, and adjusted to produce a blue flame 25 mm high. The flame is to be obtained by adjusting the gas supply and the air ports of the burner until a 25 mm yellow tipped blue flame is produced and then the air supply is to be increased until the yellow tip disappears. The height of the flame is to be measured again and corrected if necessary. The flame is to be applied to the free end at the lower edge of the specimen. The center axis of the burner tube is to be in the same vertical plane as the longitudinal bottom edge of the specimen (and inclined toward the end of the specimen) at an angle of approximately 45° to the horizontal (*see* Fig. 5). The flame is to be applied so that the front edge of

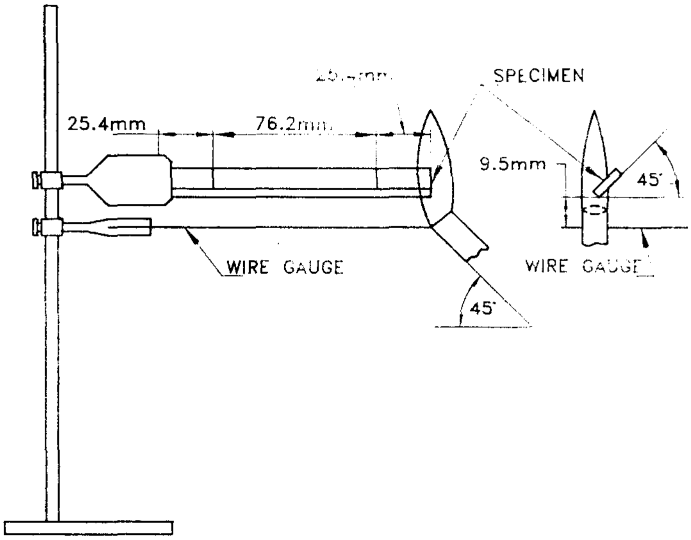


Fig. 5 Horizontal Burning Test for 94 HB Classification

the specimen, to a depth of approximately 6.4 mm, is subjected to the test flame for 30 s without changing the position of the burner, and is then removed from the specimen. If the specimen burns to the 25.4 mm mark before the flame has been applied for 30 s, the flame application is to be discontinued when the flame reaches the 25.4 mm mark.

C-2.12 If the specimen continues to burn after removal of the test flame, the time for the flame front to travel from the mark 25.4 mm from the free end to the mark 102 mm from the free end, is to be determined and the rate of burning is to be calculated.

C-3 VERTICAL BURNING TEST FOR CLASSIFYING MATERIALS 94V-0, 94V-1 OR 94V-2

C-3.1 Test Criteria

Materials shall be classified 94V-0, 94V-1 or 94V-2 on the basis of results obtained on small bar specimens when tested as described in **C-3.6** to **C-3.15**.

C-3.1.1 Some materials, due to their thinness, distort, shrink, or are consumed up to the holding clamp when subjected to this test. These materials may be tested according to the vertical burning test for classifying materials 94VTM-0, 94VTM-1 or 94VTM-2, provided specimens can be properly formed.

C-3.2 Materials Classed 94V-0

A material classed 94V-0 shall (*see also* **C-3.5**):

a) not have any specimens that burn with flaming combustion for more than 10 s after either application of the test flame.

b) not have a total flaming combustion time exceeding 50 s for the 10 flame applications for each set of five specimens.

c) not have any specimens that burn with flaming or glowing combustion up to the holding clamp.

d) not have any specimens that drip flaming particles that ignite the dry absorbent surgical cotton located 305 mm below the test specimen.

e) not have any specimens with glowing combustion that persists for more than 30 s after the second removal of the test flame.

C-3.3 Materials Classed 94V-1

A material classed 94V-1 shall (*see also* **C-3.5**):

a) not have any specimens that burn with flaming combustion for more than 30 s after either application of the test flame.

b) not have a total flaming combustion time exceeding 250 s for the flame applications for each set of five specimens.

c) not have any specimens that burn with flaming or glowing combustion up to the holding clamp.

d) not have any specimens that drip flaming particles that ignite the dry absorbent surgical cotton located 305 mm below the test specimen.

e) not have any specimens with glowing combustion that persists for more than 60 s after the second removal of the test flame.

C-3.4 Materials Classed 94V-2

A material classed 94V-2 shall (*see also* **C-3.5**):

a) not have any specimens that burn with flaming combustion for more than 30 s after either application of the test flame.

b) not have a total flaming combustion time exceeding 250 s for the 10 flame applications for each set of five specimens.

c) not have any specimens that burn with flaming or glowing combustion up to the holding clamp.

d) be permitted to have specimens that drip flaming particles that burn only briefly, some of which ignite the dry absorbent surgical cotton placed 305 mm below the test specimen.

e) not have any specimens with glowing combustion that persists for more than 60 s after the second removal of the test flame.

C-3.5 If only one specimen from a set of five specimens does not comply with the requirements, another set of five specimens is to be tested. In the case of the total number of seconds of flaming, an additional set of five specimens is to be tested if the totals are in the range of 51-55 s for 94V-0 and 251-255 s for 94V-1 and 94V-2. All specimens from this second set shall comply with the appropriate requirements in order for the material in that thickness to be classified 94V-0, 94V-1 or 94V-2.

C-3.6 Apparatus

The apparatus employed is to consist of the following:

i) *Test Chamber* — enclosure or laboratory hood free of induced or forced draft during tests.

ii) *Laboratory Burner* — A Bunsen or Tirrill burner having a tube with a length of 80-100 mm and an inside diameter of $9.4^{+1.6}_{-0.0}$ mm. The tube is not to be equipped with an end attachment, such as a stabilizer.

iii) *Ring Stand* — A ring stand with clamps or the equivalent, adjustable for vertical positioning of specimens.

iv) *Gas Supply* — A supply of technical grade methane gas with regulator and meter for uniform gas flow. Natural gas having a heat content of approximately 37 MJ/m^3 has been found to provide similar results. However, technical grade methane gas is to be used in case of dispute. Other fuel gases, such as butane, propane, and acetylene have a higher heat contact and are not acceptable.

v) Stopwatch or other timing device.

vi) A supply of dry absorbent surgical cotton.

vii) A desiccator containing anhydrous calcium chloride.

viii) Conditioning room or chamber capable of being maintained at 27 ± 2°C and a relative humidity of 65 ± 5 percent.

ix) *Conditioning Oven* — A full draft air-circulating oven capable of being maintained at 70 ± 1°C.

C-3.7 Test Specimens

Test specimens, 127 mm in length by 12.7 mm in width in the minimum and maximum thicknesses are to be tested covering the thickness range to be considered. Specimens tested by this method are limited to a maximum thickness of 12.7 mm. Specimens in intermediate thicknesses are also to be provided and may be tested if the results obtained on the minimum or maximum thickness indicate a need. Intermediate thicknesses are not to exceed increments of 3.18 mm. The specimens are to comply with the following:

a) The maximum width is to be 13.2 mm.

b) The edges are to be smooth and the radius on the corners is not to exceed 1.27 mm.

C-3.8 If the material is to be considered in a range of colours, melt flows, or reinforcements, specimens representing those ranges are also to be provided. Specimens in the natural (if used in this colour) and in the most heavily pigmented light and dark colours are to be provided and considered representative of the colour range, if the burning characteristics are essentially the same. An additional set of specimens is to be provided in the heaviest organic pigment loading, unless the most heavily pigmented light and dark colours include the highest organic pigment level. When certain colour pigments (for example, red, yellow, or the like) are known by experience to have particularly critical effects, they are also to be provided. Specimens in the extremes of the melt flows and reinforcement contents are to be provided and considered representative of the range, if the burning characteristics are essentially the same. If the burning characteristics are not essentially the same for all specimens representing the range, evaluation is to be limited only to the material in the colours, or additional specimens in intermediate colours, melt flows, and reinforcement contents are to be provided for tests.

C-3.9 Specimen Conditioning

Specimen sets are to be conditioned as follows:

i) Sets of five specimens each are to be conditioned for at least 48 h at a temperature of 27 ± 2°C and a relative humidity of 65 ± 5 percent prior to testing.

ii) Sets of five specimens each are to be conditioned in an air-circulating oven for 168 h at 70 ± 1°C and then cooled in a desiccator, over anhydrous calcium chloride, for atleast 4 h at room temperature prior to testing.

Exception: As an alternative to 168 h at 70 ± 1°C, industrial laminates may be conditioned for 24 h at 125 ± 1°C.

C-3.10 Test Method

The burning test is to be conducted in a chamber, enclosure, or laboratory hood free of induced or forced draft. An enclosed laboratory hood with a heat-resistant glass-window and an exhaust fan for removing the products of combustion after the test is recommended.

C-3.11 Each specimen is to be supported from the upper 6.4 mm of the specimen, with the longitudinal axis vertical, by the clamp of the ring stand so that the lower end of the specimen is 9.5 mm above the top of the burner tube and 305 mm above a horizontal layer of dry absorbent surgical cotton. To form the horizontal layer, a small portion, approximately 12.7 × 25.4 mm of cotton is to be pulled from the supply with thumb and forefinger and then thinned and spread with the fingers into a 5.08 mm square having a freestanding thickness of 6.4 mm.

C-3.12 The burner is then to be placed remote from the specimen, ignited and adjusted to produce a blue flame 19 mm high. The flame should be obtained by adjusting the gas supply and the air ports of the burner until a 19 mm yellow-tipped blue flame is produced and then an increase in the air supply is to be made until the yellow-tip disappears. The height of the flame is to be measured again and corrected, if necessary.

C-3.13 The test flame is to be placed centrally under the lower end of the test specimen and allowed to remain for 10 s. The test flame is then to be withdrawn at least 152 mm away and the duration of flaming of the specimen noted. When flaming of the specimen ceases, the test flame is to be immediately placed again under the specimen. After 10 s, the test flame is again to be withdrawn, and the duration of flaming and glowing is to be noted.

C-3.14 If the specimen drips molten or flaming material during either flame application, the burner may be tilted to an angle up to 45° and also slightly withdrawn from one of the 12.7 mm sides of the specimen during the flame application, to avoid material dripping into the tube of the burner. If the specimen drips molten or flaming material or is consumed during the test, the burner is to be hand-held, and the 9.5 mm distance between the bottom of the specimen and the top of the burner tube is to be maintained during the flame application. Any molten strings of the material are to be ignored, and the flame is to be applied to the major portion of the specimen.

C-3.15 The following are to observed and recorded:

a) Duration of flaming after first flame application.

b) Duration of flaming after second flame application.

c) Duration of flaming plus glowing after second flame application.

d) Whether or not specimens burn up to the holding clamp.

e) Whether or not specimens drip flaming particles that ignite cotton swatch.

C-4 VERTICAL BURNING TEST FOR CLASSIFYING MATERIALS 94-5V

C-4.1 Test Criteria

A material shall be classified 94-5V on the basis of test results obtained on small bar specimens when tested as described in **C-4.5** to **C-4.14**.

C-4.2 A material classed 94-5V shall (*see also* **C-4.4**):

i) not have any specimens that burn with flaming or glowing combustion for more than 60 s after the fifth flame, and

ii) not have any specimens that drip any particles.

C-4.3 In conjunction with **C-4.2** observation shall be made for destruction of the specimens during and after tests. If any specimens are observed to undergo shrinkage, elongation, melting or the like, additional tests shall be conducted on 152 mm × 152 mm plaques of the same thickness, in accordance with **C-4.15** to **C-4.20**. Tests of plaques shall also comply with **C-4.2**.

C-4.4 If only one specimen from a set of five specimens does not comply with the requirements, another set of five specimens are to be tested. All specimens from this second set are to comply with the requirements in order for the material in that thickness to be considered acceptable.

C-4.5 Apparatus

The apparatus employed is to consist of the following:

a) *Test Chamber* — enclosure, or laboratory hood free of induced or forced draft during tests.

b) *Laboratory Burner* — A Tirrill burner having a tube with a length of 80-100 mm and an inside diameter of $9.4^{+1.6}_{-0.0}$ mm. The tube shall not be equipped with an end attachment, such as stabilizer.

c) *Ring Stand* — A ring stand with clamps or the equivalent, adjustable for vertical positioning of specimens.

d) *Gas Supply* — A supply of technical grade methane gas with regulator and meter for uniform gas flow. Natural gas having a heat content of approximately 37 MJ/m^3 has been found to provide similar results. However, technical grade methane gas is to be used in case of dispute. Other fuel gases, such as butane, propane and acetylene have a higher heat content and are not acceptable.

e) *Mounting Block* — A block capable of positioning the burner at an angle of 20° from the vertical.

f) Stopwatch or other timing device.

g) A desiccator containing anhydrous calcium chloride.

h) Conditioning room or chamber capable of being maintained at 27 ± 2°C and a relative humidity of 65 ± 5 percent.

j) *Conditioning Oven* — A full draft air-circulating oven capable of being maintained at 121 ± 1°C.

C-4.6 Test Specimens and Plaques

Test specimens, 127 mm in length × 12.7 mm in width and test plaques approximately 152 mm × 152 mm are to be tested in a minimum and maximum thicknesses covering the thickness range to be considered. The specimens and plaques tested by this method are limited to a maximum thickness of 12.7 mm. Specimen and plaques in intermediate thicknesses are also to be provided and may be tested if the results obtained on the minimum or maximum thickness indicate a need. Intermediate thicknesses are not to exceed increments of 3.18 mm. The specimens and plaques are to comply with the following:

a) The maximum specimen width is to be 13.2 mm.

b) The edges are to be smooth and the radius on the corners is not to exceed 1.27 mm.

C-4.7 If the material is to be considered in a range of colours, melt flows, or reinforcement, specimens and plaques representing those ranges are also to be provided. Specimens and plaques in the natural (if used in this colour) and in the most heavily pigmented light and dark colour are to be provided and be considered representative of the colour range, if the burning characteristics are essentially the same. An additional set of specimens is to be provided in the heaviest organic pigment loading, unless the most heavily pigmented light and dark colours include the highest organic pigment level. When certain colour pigments (for example, red, yellow or the like) are known by experience to have particularly critical effects, they are also to be provided. Specimens and plaques in the extremes of the melt flows and reinforcement contents are to be provided and considered representative of the range, if the burning characteristics are essentially the same. If the burning characteristics are essentially the same for all specimens and plaques representing the range, evaluation is to be limited only to the materials in the colours, melt flows, and reinforcement contents tested, or additional specimens and plaques in intermediate colours melt flows, and reinforcement contents are to be provided for tests.

C-4.8 Specimen and Plaque Conditioning

The sets of five specimens and/or plaques are to be conditioned as follows:

a) Each set is to be conditioned for at least 48 h at a temperature of 27 ± 2°C and a relative humidity of 65 ± 5 percent prior to testing.

b) Each set is to be conditioned in a circulating air oven for a duration of 60 days at 121 ± 1°C and then cooled in a desiccator over anhydrous calcium chloride for at least 4 h at room temperature prior to testing. Other temperatures and/or exposures may be selected based on application.

C-4.9 Method A, Test of Specimens

The burning test is to be conducted in a chamber, enclosure, or laboratory hood free of induced or forced draft. An enclosed laboratory hood with a heat resistant glass window and an exhaust fan from removing the products of combustion after the test is recommended.

C-4.10 The test specimen is to be supported from the upper 6.4 mm of the specimen, with the longitudinal axis vertical, by the clamp on the ring stand. The burner is to be supported on the inclined plane of a mounting block so that the burner tube may be positioned at 20° from the vertical. The narrow edge of the specimen is to be face the burner (*see* Fig. 6).

C-4.11 The burner is to be placed remote from the specimen, ignited, and in a darkened room, adjusted so that when the burner is in a vertical position, the overall height of the flame is 127 mm, and the height of the inner blue cone is 38 mm (*see* Fig. 6).

C-4.12 The flame is then to be applied to one of the lower corners of the specimen at an angle of 20° from the vertical, so that the tip of the blue cone touches the specimen (*see* Fig. 6).

C-4.13 The flame is to be applied for 5 s and removed for 5 s. The operation is to be repeated until the specimen has been subjected to five applications of the test flame.

C-4.14 After the fifth removal of the test flame, the following are to be observed and recorded:

a) Duration of flaming plus glowing.

b) The distance the specimen burned or was affected.

c) Whether or not specimen dripped particles during test.

d) Observations of deformation and physical strength immediately after burning and when cooled.

C-4.15 Method B, Test of Plaques

The burning test is to be conducted in a chamber, enclosure, or laboratory hood free of induced or forced draft. An enclosed laboratory hood with a heat resistant glass window and an exhaust fan for removing the products of combustion after the test is recommended.

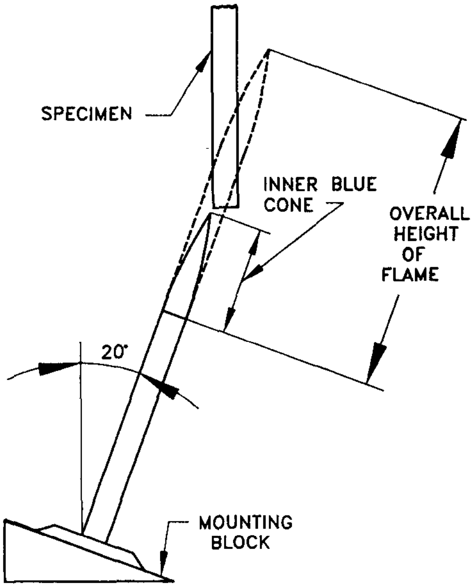


Fig. 6 Vertical Burning Test for 94-5V Classification

C-4.16 The plaques are to be tested in various positions so that the test flame is applied as follows:

a) Plaque vertical with the flame applied to lower corner of the plaque.

b) Plaque vertical with the flame applied to lower edge of the plaque.

c) Plaque vertical with the flame applied to center of one side of the plaque.

d) Plaque horizontal with the flame applied to the center of the bottom surface of the plaque.

e) Plaque horizontal with the flame directed downward to the top surface of the plaque.

C-4.17 The burner is to be placed remote from the specimen, ignited, and adjusted so that when the burner is in a vertical position, the overall height of the flame is 127 mm, and the height of the inner blue cone is 38 mm (*see also* **C-4.11**).

C-4.18 The flame is then to be applied to the test plaques in each of the positions given in **C-4.16** at an angle of 20° from the vertical, so that the tip of the blue cone touches the specimen.

C-4.19 The flame is to be applied for 5 s and removed for 5 s. The operation is to be repeated until the plaque has been subjected to five applications of the test flame.

C-4.20 After the fifth removal of the test flame, the following are to be observed and recorded:

a) Duration of flaming plus glowing.

b) The distance the plaque burned or was affected.

c) Whether or not particles dripped from the plaque during the test.

d) Observations of deformation and physical strength immediately after burning and when cooled.

ANNEX D

(*Clause* 3.2.5)

TEST METHOD FOR HAZE AND LUMINOUS TRANSMITTANCE OF TRANSPARENT PLASTICS

D-1 GENERAL

D-1.1 This test method covers the measurement of the light-transmitting properties, and from these, the light-scattering properties, of planar sections of transparent plastics. Two procedures are covered as follows.

D-1.1.1 *Procedure A* — Using the Hazemeter.

D-1.1.2 *Procedure B* — Using the Recording spectrophotometer.

NOTE — The test method for measuring luminous transmittance in Procedure A is a simplified procedure which should give results not significantly different from those obtainable if the light source were CIE Source A or C (specified) and the receptor had a spectral response corresponding to the luminosity function y of the CIE standard observer.

D-1.2 Specimens with haze values greater than 30 percent obtained by these test methods should be considered diffusing or translucent.

NOTE — When materials scatter a high percentage of light within a narrow forward angle, such as occurs with abraded transparent plastics, much of this light will be collected by the light trap and, an erroneously low value will be measured. When such materials are measured, it is recommended that the hazemeter be adjusted.

D-2 TERMINOLOGY

D-2.1 Definitions

D-2.1.1 *Haze,* that percentage of transmitted light which in passing through the specimen deviates from the incident beam by forward scattering. For the purpose of this test method only light flux deviating more than 2.5° on the average is considered to be haze.

D-2.1.2 *Luminous transmittance* is defined as the ratio of transmitted to incident light.

D-3 SIGNIFICANCE AND USE

D-3.1 The significance of the haze results obtained improves with the avoidance of heterogeneous surface and internal defects which can contribute to the diffusion or deviation of light. With this precaution and careful calibration of the apparatus, results may be obtained with a precision of ± 0.1 percentage haze. Differences of this order are rarely detectable visually. In accordance with the definition as given the accuracy of test results obtained by different operators and equipment is of the order of ± 0.3 percentage haze. Although these results are empirical, they are of value for control purposes and may be related to more fundamental properties useful in research.

D-4 CIE SOURCES

D-4.1 CIE sources A or C shall be provided by suitable filters if Procedure A is used, or by calculation if Procedure B used. Use CIE source C, unless there is reason to report the results for another CIE source.

NOTE — Standard Illuminant C is intended to represent daylight. Originally defined as source C, prepared by using source A (incandescent lamp light,colour temperature 2 856 K) with a blue liquid filter. Except in the UV-region, the spectral power distribution agrees well with that of typical daylight. The correlated colour temperature is 6 700 K. (CIE standards made available from US. National Committee, CIE, National Institute of Standards and Technology, Gaithersbung, MD 20899).

D-5 TEST SPECIMEN

D-5.1 The test specimen shall be large enough to cover a 2.5 × 2.5 cm apperture but small enough to be tangent to the sphere wall; a disk 3.5 cm in diameter is suggested. The specimen shall have substantially plane-parallel surfaces free of dust, grease, scratches, blemishes, and visibly distinct internal voids and particles, unless it is specifically desired to measure the contribution to haze due to these imperfections. Surface defects may be eliminated by immersing the specimen in a clear liquid of the same refractive index.

D-6 CONDITIONING

D-6.1 Condition the test specimens at 27 ± 2°C and 65 ± 5 percent relative humidity for not less than 40 h prior to test, for those tests where conditioning is required. In cases of disagreement, the tolerances shall be ± 1°C and ± 2 percent relative humidity.

D-6.2 Test Conditions

Conduct tests in the standard laboratory atmosphere of 27 ± 2°C and 65 ± 5 percent relative humidity, unless otherwise specified in the test methods. In cases of disagreements, the tolerances shall be ± 1°C and ± 2 percent relative humidity.

D-7 PROCEDURE A — USING THE HAZEMETER APPARATUS

D-7.1 Apparatus

The hazemeter shall be constructed essentially as shown in Fig. 7 or Fig. 8 and shall conform to the requirements in **D-7.2** to **D-7.9**.

D-7.2 An integrating sphere shall be used to collect transmitted flux; the sphere may be of any diameter so long as the total port area does not exceed 4.0 percent of the internal reflecting area of the sphere.

D-7.3 Figure 7 and Fig. 8 indicate possible arrangements of the apparatus. These contrast with that of Procedure B in that specimen and standard beams are the same beam. The entrance and exit ports shall be centered on the same great circle of the sphere and there shall be at least 170° of arc between centres. The exit port shall subtend an angle of 8° at the center of the entrance port. The axis of the irradiating beam shall pass through the centers of the entrance and exit ports. The photocell or photocells shall be positioned on the sphere 90 ± 10° from the entrance port. In the pivotable modification of this type (Fig. 8) designed to use the interior sphere wall adjacent to the exit port as the reflectance standard, the angle of rotation shall not exceed 10°.

D-7.4 The specimen shall be illuminated by a substantially unidirectional beam; the maximum angle

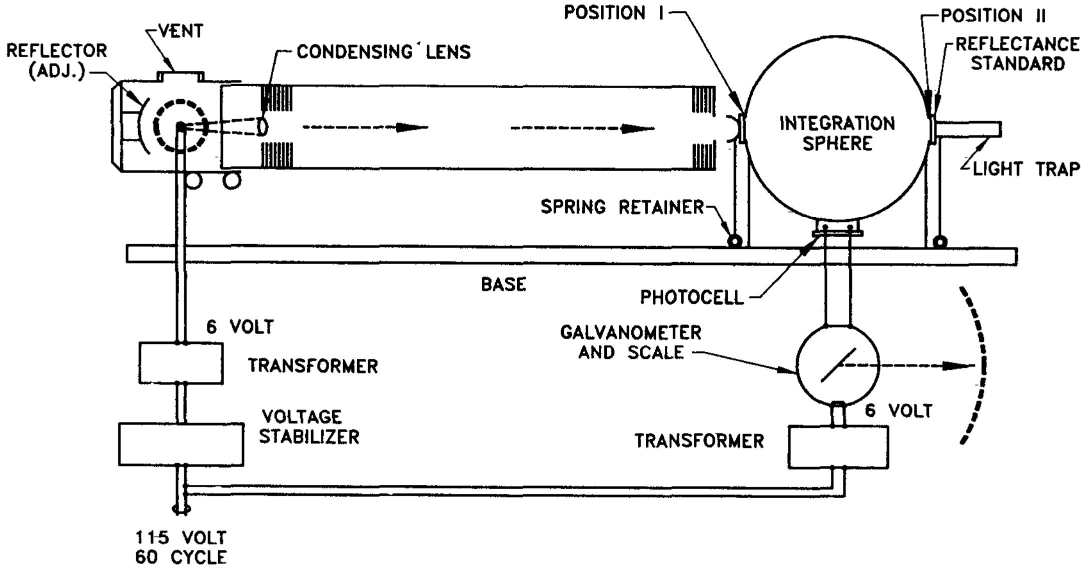
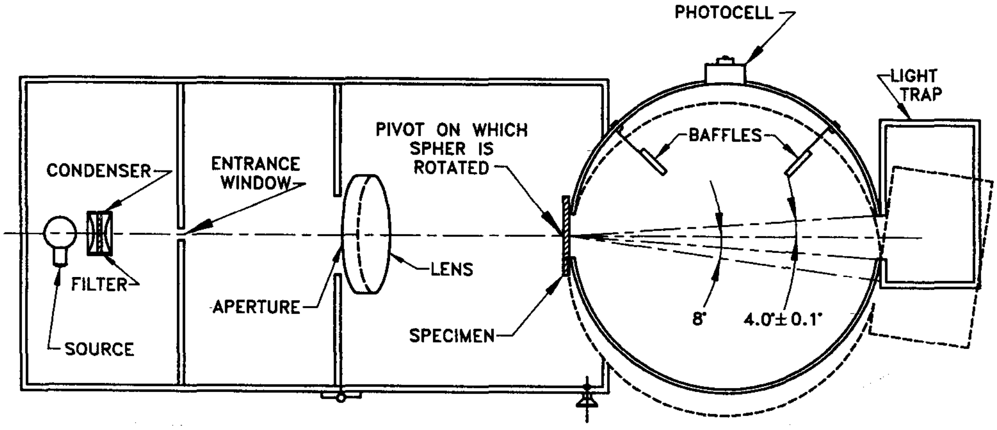


FIG. 7 HAZEMETER



NOTE — Dotted lines show position of sphere for total transmission measurements.

FIG. 8 PIVOTABLE-SPHERE HAZEMETER

which any ray of this beam makes with the direction of its axis shall not exceed 3°. This beam shall not be vignetted at either port of the sphere.

D-7.5 When the specimen is placed immediately against the integrating sphere at the entrance port, the angle between the normal to its surface and the axis of the beam shall not exceed 8°.

D-7.6 When the beam is unobstructed by a specimen, its cross-section at the exit port shall be approximately circular, sharply defined and concentric within the exit port, leaving an annulus of 1.3 ± 0.1° subtended at the entrance port.

NOTE — It is important to check the unobstructed-beam annulus dimensions whenever apertures or focus are changed.

D-7.7 The surfaces of the interior of the integrating sphere, baffles, and reflectance standards shall be of substantially equal reflectance, matte, and highly reflecting throughout the visible wave lengths.

NOTE — Freshly smoked magensium oxide in excellent for this purpose but highly reflecting matter sphere paints are more durable.

D-7.8 For some measurements the standard at the exit port is replaced by a light trap by actual removal of the reflectance standard or by pivoting the sphere (Fig. 8). The light trap shall absorb the beam completely when no specimen is present.

NOTE — Due to the absorbing annulus surrounding the unimpeded beam at the exit port, this trap will absorb slightly more than the undeviated portion of the total flux transmitted by the specimen.

D-7.9 The radiant flux within the sphere shall be measured by a photoelectric cell, the output measurements of which shall be proportional within 1 percent to the incident flux over the range of intensity used. Spectral conditions for source and receiver must be constant throughout the test of each specimen. The design of the instrument shall be such that there shall be no galvanometer deflection when the sphere is dark.

D-8 PROCEDURE

D-8.1 Determine the following four readings:

Reading Designation	*Specimen in Position*	*Light Trap in Position*	*Reflectance Standard in Position*	*Quantity Represented*
T_1	No	No	Yes	Incident light
T_2	Yes	No	Yes	Total light transmitted by specimen
T_3	No	Yes	No	Light scattered by instrument
T_4	Yes	Yes	No	Light scattered by instrument and specimen

D-8.2 Repeat readings for T_1, T_2, T_3 and T_4 with additional specified positions of the specimen to determine uniformity.

D-9 CALCULATION

D-9.1 Calculate total transmittance, T_t (Note), equal to T_2/T_1.

D-9.2 Calculate diffuse transmittance T_d (Note), as follows:

$$T_d = \frac{T_4 - T_3\,(\,T_2/T_1\,)}{T_1}$$

D-9.3 Calculate percentage haze as follows:

$$\text{Haze, percent} = \frac{T_d}{T_t} \times 100$$

NOTE — To obtain luminous transmittance values (total T_t diffuse, T_d) by this procedure, a proper source filter-photocell system must be employed. A Western Type III photocell, with Viscor Filter, approximates the response of the standard observer except at the extremes of the visible spectrum. The source may be operated at a colour temperature approximating 2 854 K (CIE Source A) by proper selection of voltage. CIE Source C may be achieved with CIE Source A and a filter described by Davis. R. and Gibson, K.S., *Miscellaneous Publication No. 114*, Nat. Bureau Standards, January 1931. An approximation of CIE Source C may be obtained with the lamp operating at 3 100 K plus a Corning Type 5900 filter of standard thickness.

Commercially produced hazemeters are equipped to provide luminous transmittance measurements, using either CIE Source.

To obtain the greatest accuracy in luminous transmittance measurement when using a single-beam instrument, it is necessary to use a standard, calibrated with a double-beam instrument. Otherwise, insertion of the sample in the single-beam instrument changes the efficiency of the sphere. This change may result in spuriously high readings for clear, colourless samples and significant errors for dark or highly saturated colours. In these cases, the photometer should be used as a comparison instrument with a standard of known transmittance similar to that of the specimen.

D-10 PROCEDURE B — USING THE RECORDING SPECTROPHOTOMETER

D-10.1 Apparatus

The recording spectrophotometer shall conform to the requirements in **D-10.2** to **D-10.10**.

D-10.2 An integrating sphere shall be used to collect transmitted flux. The sphere may be of any diameter so long as the total area of the ports does not exceed 4.0 percent of the internal reflecting area of the sphere.

D-10.3 Emitted by one source, specimen and standard beams shall be deviated by a small angle so that they enter the sphere as two distinct beams through different entrance ports. These ports shall be located on the surface of the sphere, on a horizontal line and equidistant from a line from point of deviation to the center of the sphere. The angle of deviation and the planes of the exit port shall be oriented so that the unobstructed beams at these ports are approximately circular.

D-10.4 The test specimen shall be illuminated by a substantially unidirectional beam; the maximum angle which any element of this beam makes with the direction of its axis shall not exceed 3°. This beam shall not be vignetted at either port of the sphere.

D-10.5 When the test specimen is placed immediately against the integrating sphere at the specimen entrance port, the angle between the normal to its surface and the axis of the sample beam shall not exceed 8°.

D-10.6 When the specimen beam is unobstructed by a specimen, its cross-section at the exit port must be approximately circular, sharply defined, and concentric within the port, leaving an annulus of 1.3 ± 0.1°.

D-10.7 The surfaces of the interior of the integrating sphere, baffles, and reflectance standards shall be substantially equal in reflectance, matte and highly reflecting throughout the visible wavelengths.

D-10.8 When diffuse luminous transmittance T_d is determined, the standard at the specimen exit port is replaced by a light trap which shall absorb the specimen beam when unobstructed by a specimen.

D-10.9 The radiant flux within the sphere shall be measured by a photoelectric cell as the flux transmitted by either of the following two acceptable methods.

D-10.9.1 The flux may be transmitted by a highly diffusing translucent window of opal glass so located in the sphere that it becomes a continuous portion of its interior surface. The photocell port is located at the lower pole of the vertical diameter. If the flux is of such low magnitude that insufficient sensitivity is obtained, improvement will result if the opal glass window is removed. If this is done, a baffle of similar translucency, for example, high quality bond paper, must be located on the interior surface of the sphere between photocell and exit port, to prevent direct reflection from the reflectance standards to the photoelectric cell.

D-10.9.2 The flux may be transmitted by a clear acrylic plastic rod extending up from the bottom of the sphere to just above the center between the two beams. This pipes the flux down to the phototube. It also prevents direct reflection from the reflectance standards to the photoelectric cell.

D-10.10 Values of spectral diffuse transmittance may be quite low; they must be recorded and integrated carefully. Improved accuracy will result from the use of a mechanically introduced 5 × factor, applicable for curves that are less than 20 percent at all wavelengths. If a multiplying factor is not used, improved accuracy can be obtained by employing a correlation between indicated photometric readings and transmittance values in this range. The operating instructions of the instrument manufacturer must be followed closely and particular attention must be given to the zero adjustment of the recording system.

NOTE — These requirements are met by a recording spectrophotometer of the Hardy type. There are two different types of integrating spheres in general use with this instrument, as illustrated in Fig. 9 and Fig. 10. The principal difference is one of geometry. In the early or "diffuse (reflectance)" type the unobstructed light beams meet the reflectance standards normal to their surfaces, as shown in Fig. 7. In the later or "specular (reflectance)" type the axes of these beams meet the reflectance standard at an angle of 6° from the normal, as shown in Fig. 10.

D-11 PROCEDURE

D-11.1 Check the wavelength and photometric scales of the instrument.

NOTE — One method of making wavelength corrections is by running the curve of a calibrated didymium glass. On the general electric Hardy type instrument the photometric scale can be checked by running 100 percent and zero curves and if desirable by running transmittance curves of standard glasses or solutions.

D-11.2 Determine whether or not specimens are birefringent by rotating them between crossed Polaroids or other polarizer and analyzer. Transmittance change with rotation indicated birefringence. Measure birefringent specimens with polarized light in such a manner that the results obtained are valid for unpolarized light.

D-11.3 Place the sample directly in contact with the entrance port of the sphere for transmittance measurements.

NOTE — Direct contact with the entrance port is necessary if all of the scattered light is to enter the sphere. To accomplish this with spheres having a connecting sleeve between sphere and sample compartment, a small specimen may be used, or a modification may be made in the instrument. One modification consists of removing a portion of the connecting sleeves on the specimen side, between the integrating sphere and the sample compartment. A portion of the specular cup and associated sphere must be removed to permit proper positioning of the specimen.

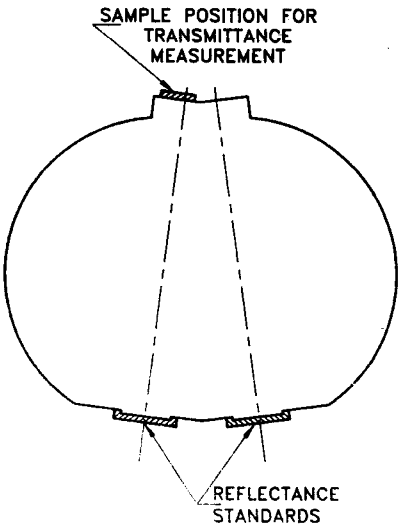


FIG. 9 INTEGRATING SPHERE FOR RECORDING SPECTROPHOTOMETER 'DIFFUSE (REFLECTANCE)' TYPE

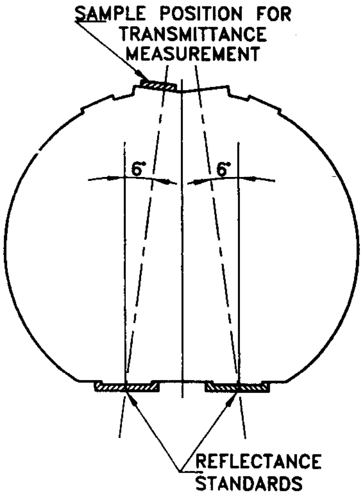


FIG. 10 INTEGRATING SPHERE FOR RECORDING SPECTROPHOTOPMETER, 'SPECULAR (REFLECTANCE)' TYPE

D-11.4 Record the following four curves:

Curve Designation	*Specimen in Position*	*Light Trap in Position*	*Reflectance Standard in Position*	*Quantity Represented*
C_1	No	No	Yes	Incident light
C_2	Yes	No	Yes	Total light transmitted by specimen
C_3	No	Yes	No	Light scattered by instrument
C_4	Yes	Yes	No	Light scattered by instrument and specimen

D-11.5 Repeat curves C_2 and C_4 above with different positions of specimen to determine uniformity. Recommended additional positions are:

D-11.5.1 Specimen rotated 90° in plane parallel to its surfaces.

D-11.5.2 Specimen reversed so that light beam is incident upon surface which was next to sphere.

D-11.5.3 Specimen rotated 90° from position (*see* **D-11.5.2**) in plane parallel to its surfaces.

D-12 CALCULATION

D-12.1 Obtain the values of T_1, T_2, T_3 and T_4 by either of the following two acceptable methods:

D-12.1.1 For the recorded curves, compute T_1, T_2, T_3 and T_4 by integration of CIE Sources A and C and the luminosity function y. A sufficient number of ordinates shall be used so that increasing their number produces a difference not exceeding 0.1 percent haze. An automatic digital read-out device, connected directly to the recording spectrophotometer, may be used to obtain transmittances at stated wavelength intervals, eliminating the necessity of obtaining these values from the recorded curves.

D-12.1.2 Obtain the values T_1, T_2, T_3 and T_4 by the use of an automatic integrator which is connected directly to the spectrophotemeter, and which calculates the values accurately for the specified CIE Source.

D-12.2 Calculate total luminous transmittance, T_t, equal to T_2/T_1.

D-12.3 Calculate diffuse luminous transmittance as follows:

$$T_d = \frac{T_4 - T_3\,(T_2/T_1)}{T_1}$$

D-12.4 Calculate percentage haze as follows:

Haze, percent = $(T_d/T_t) \times 100$

D-13 DERIVATION OF FORMULAS FOR HAZE

D-13.1 The derivation of the formula for haze for both procedures is as follows:

D-13.1.1 Total luminous transmittance, T_t, is calculated as follows:

$$T_t = T_2/T_1$$

where

T_2 = total light transmitted by the specimen, and

T_1 = incident light.

D-13.1.2 If T_3, the light scattered by the instrument, is zero, the diffuse luminous transmittance, T_d, is calculated as follows:

$$T_d = T_4/T_1$$

where

T_4 = light scattered by the instrument and specimen.

D-13.1.3 If T_3 is greater than zero due to light scattered by the instrument, the total scattered light, T_4, will be greater than the light scattered by the specimen by an amount proportional to T_3 and equal to T_3 times T_2/T_1. The corrected amount of light scattered by the specimen will then be:

$$T_4 - T_3\,(T_2/T_1)$$

D-13.1.4 The diffuse luminous transmittance, T_d, is then calculated as follows:

$$T_d = \frac{T_4 - T_3\,(T_2/T_1)}{T_1}$$

D-13.1.5 Percentage haze is then calculated from the ratio of diffuse, T_d, to total luminous transmittance, T_t, as follows:

$$\text{Haze, percent} = (T_d/T_t) \times 100$$

$$= \frac{T_4 - T_3\,(T_2/T_1)}{T_1\,(T_2/T_1)} \times 100$$

$$= \frac{T_4 - T_3\,(T_2/T_1)}{T_2} \times 100$$

$$= \left(\frac{T_4}{T_2} - \frac{T_3}{T_1}\right) \times 100$$

ANNEX E

(*Clause* 5)

SAMPLING OF POLYCARBONATE MOULDING MATERIALS

E-1 GENERAL REQUIREMENTS OF SAMPLING

E-1.1 Sample shall not be taken in an exposed place.

E-1.2 The sampling instrument, which shall be made of glass, stainless steel or any other material on which polycarbonate moulding material has no action, shall be clean and dry.

E-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and containers selected for sampling from adventitious contamination.

E-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

E-1.5 The sample shall be placed in suitable, clean, dry, air-tight sheet metal or glass containers on which the material has no action.

E-1.6 Each sample container shall be sealed air-tight with a stopper after filling and marked with full details of sampling, the date of sampling and the month and year of manufacture of the material.

E-2 SCALE OF SAMPLING

E-2.1 Lot

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

E-2.2 A number of containers, consisting 10 percent of the containers in a lot but not less than 3 containers in any case, shall be selected at random from a lot for the purpose of drawing samples for test (*see* IS 4905).

E-3 TEST SAMPLES AND REFEREE SAMPLE

E-3.1 Preparation

To prepare a set of test samples, draw with an appropriate sampling instrument, from freshly opened containers which have been selected for sampling, an equal number of scoopfuls of material from any point at least 75 mm below the surface and 75 mm above the bottom of large containers, and from any point at least 25 mm below the surface and 25 mm above the bottom of small containers. The sample prepared by mixing the portions from each container shall be not less than eight times the quantity which is estimated to be required for carrying out all the tests. Divide this composite sample into the required number of reduced samples. Each set of these reduced samples shall constitute the test sample.

E-3.2 Three sets of test samples, each not less than twice the quantity required for the purpose of testing, representative of each selected container (*see* **E-3.1**) shall be transferred immediately to thoroughly dried containers, which shall be sealed airtight with an appropriate stopper. These containers shall be marked with all the particulars of sampling given under **E-1.6**. One set of the test samples shall be sent to the purchaser and one to the supplier.

E-3.3 Referee Sample

The third set of the test samples, bearing the seals of the purchaser and the supplier shall constitute the referee sample, to be used in case of dispute between the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier.

E-4 TEST FOR ACCEPTANCE

E-4.1 Examination and Tests

The purchaser may examine and test separately samples from each of the lots (*see* **E-2.1**) for compliance with the requirements of the standard, or he may prepare for the purpose of such examination, a composite sample representing the whole of the consignment, by mixing the test samples.

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